"California's Full Service Hazardous Waste Recycler"

PRC

— are they distributing the information on QA/QC to other in the industry?

Greg Czajkowski, Chief
Toxics Section
Pesticides and Toxics Branch
Air and Toxics Division
United State Environmental Protection Agency
Region 9
75 Hawthorne Street
San Francisco, CA 94105

March 18, 1992

RE: Docket No. TSCA-09-91-0002

Dear Mr. Czajkowski;

This letter is in response to the Final Consent Agreement and Final Order, page 4, item number 4.

Petroleum Recycling Corporation has adopted a QA/QC program which is defined in the attached Waste Analysis Plan. PRC has also hired a QA/QC Manager who runs the program.

PRC certifies that the Facility is being operated in full compliance with T.S.C.A. and the implementing regulations.

If you have any questions, please feel free to call.

Sincerely,

Richard D. McAuley
President

RDM/kt
Encl:

PETROLEUM RECYCLING CORPORATION
Corporate Office: 2651 Walnut Avenue • Signal Hill, CA 90806 • 213-595-7431 • *Fax: 213-427-2084*
Patterson Office: P.O. Box 1167 • Patterson, CA 95363 • 209-892-6742 • *Fax: 209-892-2248, 209-892-8574 (Plant)*

WASTE ANALYSIS PLAN

Prepared for:

PETROLEUM RECYCLING CORPORATION
SIGNAL HILL, CALIFORNIA

Prepared by:

MITTELHAUSER CORPORATION
LAGUNA HILLS, CALIFORNIA

JUNE 1991

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SECTION 1.0

INTRODUCTION

1.0 INTRODUCTION

Petroleum Recycling Corporation (PRC) is primarily a recycling facility for used oil, oily water, and oily wastes. Other activities include bulking and transferring of hazardous waste fuel and bulking waste antifreeze for offsite recycling. Other pertinent information is as follows:

Owner/Operator:	Petroleum Recycling Corporation 2651 Walnut Avenue Signal Hill, CA 90806
Facility:	Petroleum Recycling Corporation 1835 East 29th Street Signal Hill, CA 90806
EPA ID No.:	CAT080011059
Type of Facility:	Waste Oil Recycler
SIC Code:	2992

This Waste Analysis Plan (WAP) was prepared in accordance with the applicable regulations at 40 CFR 264.13 and 22 CCR 67102(b) and (c). The WAP is reviewed at least annually and updated as necessary by the laboratory manager. This WAP addresses the receipt of offsite wastes and also outlines sampling and analysis procedures for PRC-generated wastes. Section 2.0 outlines operations at the facility. Section 3.0 describes the frequency of analysis to be performed. Section 4.0 describes sampling equipment and procedures and includes diagrams. Section

5.0 identifies specific analytical methods to be used. Section 6.0 outlines procedures to comply with various regulations such as Land Ban or Benzene NESHAPS.

SECTION 2.0

FACILITY DESCRIPTION

2.0 FACILITY DESCRIPTION

PRC's Signal Hill facility receives and processes a variety of oily wastes to produce used oil fuel that is sold to the fuel market for further blending and sale. Oily water is processed through the wastewater treatment system. Individual shipments of hazardous waste fuel are bulked and blended prior to being transported to an offsite incinerator. In addition, waste antifreeze is collected in a segregated tank prior to being transported to an offsite recycler. Figure 2-1 is a process flow diagram for facility operations identifying treatment processes and wastes generated by them.

Materials may be received in tank trucks or drums. Tank trucks are unloaded into receiving tanks. Drums are either unloaded directly into tanks or moved to the container storage area and emptied later. Drums are normally emptied at Rack #2 located adjacent to the container storage area using pumps and hoses.

2.1 USED OIL OPERATIONS

Used oil is processed by chemical, thermal, and/or gravity separation in tanks. Additional treatment processes include centrifugation and thermal drying. The treatment

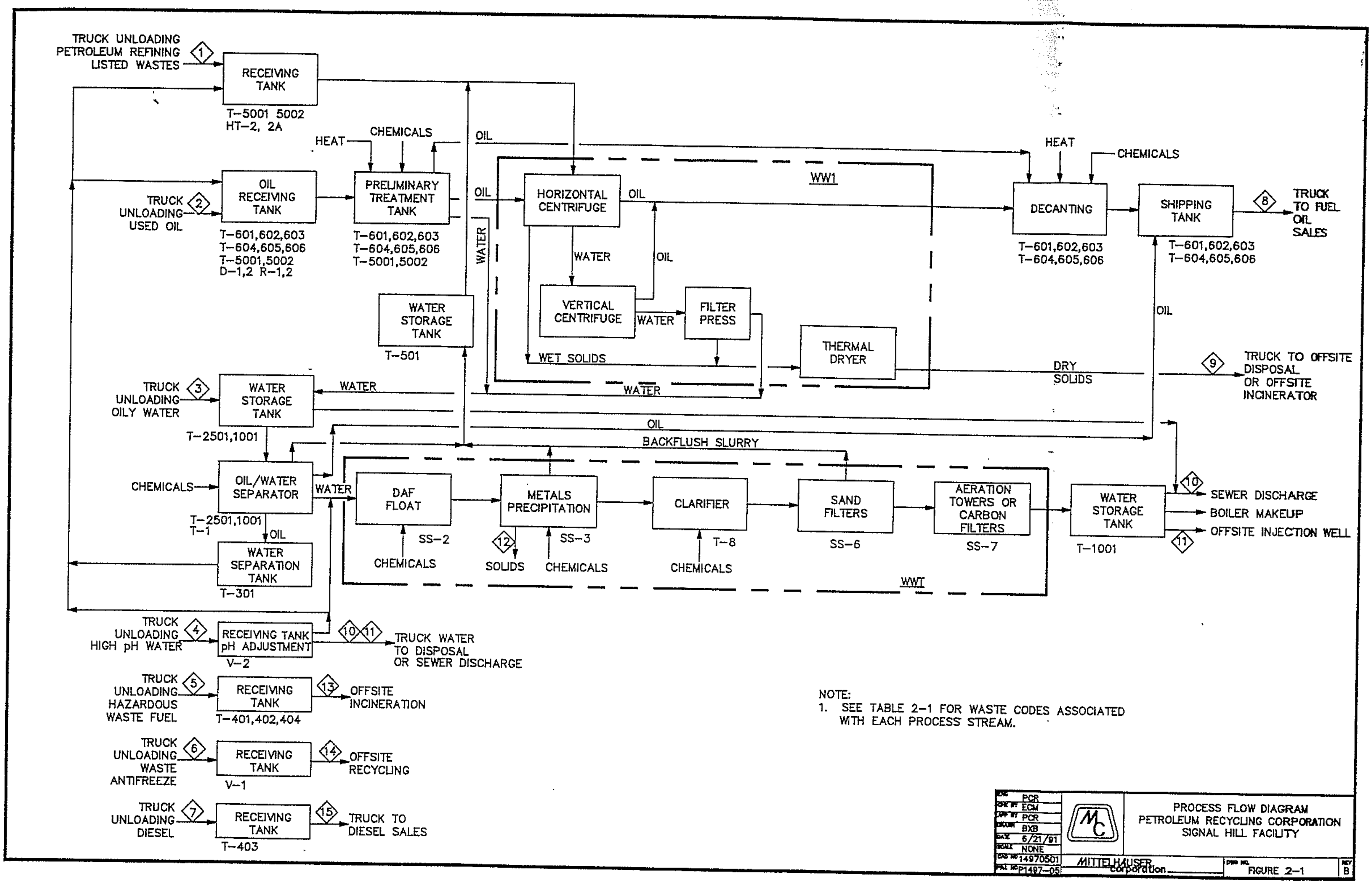

TRUCK UNLOADING PETROLEUM REFINING LISTED WASTES
1
RECEIVING TANK
T-5001 5002
HT-2, 2A
HEAT
CHEMICALS
OIL
TRUCK UNLOADING USED OIL
2
OIL RECEIVING TANK
T-601,602,603
T-604,605,606
T-5001,5002
D-1,2 R-1,2
PRELIMINARY TREATMENT TANK
T-601,602,603
T-604,605,606
T-5001,5002
WATER
WATER STORAGE TANK
T-501
HORIZONTAL CENTRIFUGE
WW1
WATER
OIL
VERTICAL CENTRIFUGE
FILTER PRESS
THERMAL DRYER
WET SOLIDS
HEAT
CHEMICALS
DECANTING
T-601,602,603
T-604,605,606
SHIPPING TANK
T-601,602,603
T-604,605,606
8
TRUCK TO FUEL OIL SALES
DRY SOLIDS
9
TRUCK TO OFFSITE DISPOSAL OR OFFSITE INCINERATOR
TRUCK UNLOADING OILY WATER
3
WATER STORAGE TANK
T-2501,1001
BACKFLUSH SLURRY
CHEMICALS
OIL/WATER SEPARATOR
T-2501,1001
T-1
WATER SEPARATION TANK
T-301
DAF FLOAT
SS-2
METALS PRECIPITATION
SS-3
12
SOLIDS
CLARIFIER
T-8
SAND FILTERS
SS-6
AERATION TOWERS OR CARBON FILTERS
SS-7
WWT
WATER STORAGE TANK
T-1001
10
SEWER DISCHARGE
BOILER MAKEUP
OFFSITE INJECTION WELL
11
TRUCK UNLOADING HIGH pH WATER
4
RECEIVING TANK pH ADJUSTMENT
V-2
10 11
TRUCK WATER TO DISPOSAL OR SEWER DISCHARGE
TRUCK UNLOADING HAZARDOUS WASTE FUEL
5
RECEIVING TANK
T-401,402,404
13
OFFSITE INCINERATION
TRUCK UNLOADING WASTE ANTIFREEZE
6
RECEIVING TANK
V-1
14
OFFSITE RECYCLING
TRUCK UNLOADING DIESEL
7
RECEIVING TANK
T-403
15
TRUCK TO DIESEL SALES
NOTE:
1. SEE TABLE 2-1 FOR WASTE CODES ASSOCIATED WITH EACH PROCESS STREAM.
PCR
ECM
PCR
BXB
6/21/91
NONE
14970501
P1497-05
MITTELHAUSER corporation
PROCESS FLOW DIAGRAM
PETROLEUM RECYCLING CORPORATION
SIGNAL HILL FACILITY
FIGURE 2-1
B

TABLE 2-1

NOTES for Process Flow Diagram

Stream #	Description of Stream	California Waste Codes	Federal Waste Codes
1	Petroleum refinery listed wastes, and high solids or high water content waste	133, 134, 135, 212, 213, 214, 221, 223, 291, 341, 342, 343	D001, D003, D004-D011, D018, D023-D025, F037, F038, K048-K052
2	Used oil	133, 134, 135, 212, 213, 214, 221, 223, 291, 341, 342, 343	
3	Oily water	133, 134, 135, 212, 213, 214, 221, 223, 291, 341, 342, 343	
4	High pH waste	121, 122, 133, 134, 135, 141, 221, 222, 223, 224	D001, D002
5	Hazardous waste fuel	211, 212, 213, 214, 221, 223, 251, 252, 291, 351, 461	D001, D003, D004-D011, D018, D019, D021, D023-D025, D035, F001-F005, K086, All U codes found in Table III-1
6	Antifreeze	331, 343	D001, D002
7	Diesel fuel		
8	Recycled oil		
9	Liquid free solids from thermal dryer	223	F037, F038, K048-K052
10	Water discharged to POTW		F037, F038, K048-K052
11	Water discharged to offsite injection well		
12	Sludge from metals precipitation unit		D004-D011 (possible), F037, F038, K048-K052
13	Hazardous waste fuel		D001, D003, D004-D011, D018, D019, D021, D023-D025, D035, F001-F005, K086, All U codes found in Table III-1.
14	Antifreeze	331, 343	D001, D002
15	Diesel fuel		

objectives are to remove impurities such as solids and water to produce a marketable used oil fuel that meets used oil specifications. Used oil tanks are sampled and analyzed at least once a day to determine whether the material meets specifications or whether additional treatment is necessary.

2.2 PETROLEUM REFINING K- AND F-LISTED WASTES

Listed hazardous wastes from the petroleum refining industry (waste codes K048, K049, K050, K051, K052, F037, and F038) may be received at the facility for treatment in the centrifuge and dryer. Treatment residuals resulting from processing include solids from the dryer, which are collected in lined roll-off bins or supersacks prior to transport to an offsite incinerator. The oil that is recovered is not a hazardous waste due to the exemptions for recyclable materials. The resultant water stream is treated in the facility wastewater treatment system prior to discharge to the Sanitation Districts of Los Angeles County.

2.3 OILY WATER

Oily water received from generators or generated onsite is treated in the wastewater treatment system. Oil recovered during treatment is returned to the used oil processing system.

Clean water is discharged to the sewer system. Sampling and analysis for discharge to the sewer are performed in accordance with the requirements of PRC's Sanitation District's permit.

Treatment residuals such as oil/water separation sludge or DAF float are treated onsite. Other wastes such as metals precipitation sludge will be sent offsite for treatment and/or disposal.

2.4 HAZARDOUS WASTE FUEL

Individual shipments of hazardous waste fuel consisting of oily wastes mixed with solvents or other listed hazardous wastes are consolidated and blended in segregated tanks prior to being transported to an offsite incinerator. Sampling and analysis are conducted to determine compliance with the receiving facility's requirements. No treatment residuals are generated because the entire volume of the tank can be sent for incineration.

2.5 WASTE ANTIFREEZE

Shipments of waste antifreeze are consolidated in Tank V-1 prior to transport to an offsite antifreeze recycler. Typical contaminants include metals.

SECTION 3.0

FREQUENCY OF SAMPLING & ANALYSIS

3.0 FREQUENCY OF SAMPLING & ANALYSIS

Each waste stream must go through a prequalification profiling procedure. Every profiled waste stream is generator-specific and a new waste from that generator would still have to be profiled. Once a waste stream has been profiled, each shipment of that waste is screened. At least one sample will be taken from every shipment that is received at PRC. If a waste stream changes or the process that generates the waste stream changes, the waste stream must be profiled again. Profiles are valid for 1 year at which time the waste stream must be profiled again. The generator may sign an identical prequalification survey if the composition of the waste or the process generating the waste has not changed. A copy of PRC's prequalification survey (profile form) is contained in Attachment 3-1.

3.1 PROFILING

Each waste stream received at the facility is profiled prior to receipt of the first shipment of the waste by the facility. Profiling activities include completion of a prequalification survey which identifies the generating process and expected composition as well as analytical procedures conducted by PRC. Analytical tests depend on the category of waste (oily water, K-wastes, alternative fuel, etc.) and the extent of information already known about the waste such as the generating process. Profiling analyses will include parameters such as PCBs, halogens, sulfides, and other parameters such as metals or cyanides, if necessary. Specific profiling parameters are determined by the laboratory manager or his designee, usually in conjunction with the marketing department, who may specifically require particular profiling parameters for pricing reasons.

Profiling activities are performed once. Subsequent shipments will be screened as outlined in Section 3.2 for conformance with profiling information. Should a waste not conform to the profiling information the waste will be reprofiled.

3.2 SCREENING

As required by the regulations, each previously profiled waste received at the facility is screened for conformance with the profile prior to acceptance by PRC. Screening activities include inspection and possibly sampling and analysis.

3.2.1 Inspection Procedures

Screening procedures are implemented after an initial inspection to determine if the waste matches the description on the Uniform Hazardous Waste Manifest. The color and odor are checked to determine if the material resembles the identification of oil, crude, etc., on the manifest.

3.2.2 Shipment Screening Analyses

Every waste shipment received at the facility is checked against the manifest. The manifest is checked to ensure that the facility is not accepting unauthorized waste codes. A visual inspection is conducted to identify any discrepancies between the material and its waste profile. Every shipment received at the facility is sampled for a "fingerprint" screening which includes, at a minimum, analysis of total organic halogen content (water) or total halogens (oil) and API gravity. Odor is checked for any solvent smell. Color and BS&W are also checked.

A maximum and minimum limit of 20 percent will be used to confirm conformance with profiling results. A waste that does not fall within the limit will be reprofiled. A waste can be accepted at the facility only if it passes the screening criteria or if reprofiling data indicates the waste is acceptable for receipt. A waste's nonconformance with its limits is documented on a rejection slip, regardless of whether the waste is reprofiled and accepted or actually turned away.

The generator is notified if a shipment cannot be accepted at the facility, and arrangements are made to transfer the waste to an appropriate facility or return it to the generator.

3.3 PROCEDURES IF CHANGE IN WASTE IS SUSPECTED

Information regarding a change in waste composition is either supplied by the generator or discovered through the inspection and screening procedures at PRC. All incoming waste shipments are sampled and analyzed. Certain key parameters are identified for each waste stream during profiling as indicator parameters for that waste stream. At a minimum, these parameters include total organic halogens or total halogens and odor. Analytical information will be computerized so that constituents that were borderline during profiling can be flagged for analysis during screening of subsequent loads.

A change in waste composition does not necessarily mean the waste cannot be accepted. If the screening analysis or other information indicates a change in the waste, the generator is contacted in order to reprofile the waste stream. The waste stream may still be acceptable if it falls within the acceptable waste codes and constituent requirements at PRC.

Whenever possible, PRC assists the generator of wastes that are unacceptable for receipt at its facility to locate an acceptable facility. If an acceptable facility is not identified, the waste is returned to the generator.

3.4 RECHARACTERIZATION OF WASTES

Reprofiling of wastes is conducted annually or at any time a change in the waste is suspected by either the generator or PRC. In lieu of retesting, the generator may sign a prequalification survey identical to the original thereby certifying that the composition or process generating the waste has not changed.

ATTACHMENT 3-1

PREQUALIFICATION SURVEY

PRE-QUALIFICATION SURVEY

PETROLEUM RECYCLING CORP.

PRC

OFFICE: 2651 Walnut Avenue, Signal Hill, California 90806 (213) 595-7431
PLANT: 1835 East 29th Street, Signal Hill, California 90806 (213) 595-6597

PQS #

APPROVAL #

A GENERATOR INFORMATION

NAME: ____________________

ADDRESS: ____________________

USEPA ID# ____________________

REPRESENTATIVE INFORMATION:

NAME: ____________________

ADDRESS: ____________________

PHONE #'S ____________________

TECHNICAL CONTACT ____________ TITLE: ____________ PHONE: ____________

PRC SALES REP: ____________

NAME OF WASTE ____________

PROCESS GENERATING WASTE ____________

B PHYSICAL CHARACTERISTICS OF WASTE (SHOW ANTICIPATED RANGES)

COLOR	ODOR	PHYSICAL STATE @ 70°F	LAYERS	FREE LIQUIDS -	BTU VALUE
	☐ NONE ☐ MILD ☐ STRONG ____	☐ SOLID ☐ SEMI-SOLID ☐ LIQUID	☐ MULTILAYERED ☐ BI-LAYERED ☐ SINGLE PHASED	☐ YES VOLUME %____	BTU/LB ____

pH ANTICIPATED RANGE	DENSITY OR API GRAVITY	FLASH POINT
☐ ≤2 ☐ 10.1 - 12.4 ☐ > 2 - 4 ☐ ≥ 12.5 ☐ 4.1 - 10 ☐ EXACT	____ ☐ LIQUID lb/gal ☐ SOLID lbs/ft	☐ < 70°F ☐ > 200°F ☐ 70°F - 100°F ☐ NO FLASH ☐ 101°F - 139°F ☐ EXACT ____ ☐ 140°F - 200°F ☐ OPEN ☐ CLOSED CUP

C CHEMICAL COMPOSITION (TOTALS MUST ADD TO 100%)

____	____ %
____	____ %
____	____ %
____	____ %
____	____ %
____	____ %
____	____ %
____	____ %
____	____ %
____	____ %

D METALS ☐ TOTAL (PPM) ☐ EPA EXTRACTION PROCEDURE (mg/L)

ARSENIC	____	SELENIUM (Se)	____
BARIUM (Ba)	____	SILVER (Ag)	____
CADMIUM (Cd)	____	COPPER (Cu)	____
CHROMIUM	____	NICKEL (Ni)	____
MERCURY (Hg)	____	ZINC (Zn)	____
LEAD (Pb)	____	THALLIUM (Tl)	____
____	____	____	____

E OTHER COMPONENTS - TOTAL (PPM)

CYANIDES ____ PCBS ____

SULFIDES ____ PHENOLICS ____

F SHIPPING INFORMATION

PROPER SHIPPING NAME ____________

HAZARD CLASS ____________ I.D.NO. ____________

METHOD OF SHIPMENT ☐ BULK LIQUID ☐ DRUM (TYPE SIZE) ____________

ANTICIPATED VOLUME ________ GALS

________ OTHER ________

PER ☐ ONE TIME ☐ WEEK ☐ MONTH ☐ QUARTER ☐ YEAR ____ ☐ ____

G HAZARDOUS CHARACTERISTICS

REACTIVITY ☐ NONE ☐ PYROPHORIC ☐ SHOCK SENSITIVE ☐ EXPLOSIVE ☐ WATER REACTIVE ☐ OTHER ____

OTHER HAZARDOUS CHARACTERISTICS

☐ NONE ☐ RADIOACTIVE ☐ ETIOLOGICAL

☐ PESTICIDE MANUFACTURING WASTE ☐ OTHER ____

USEPA HAZARDOUS WASTE? ☐ YES ☐ NO

USEPA HAZARDOUS CODE(S) ____________

STATE HAZARDOUS WASTE? ☐ YES ☐ NO

STATE CODE(S) ____________

H SPECIAL HANDLING INFORMATION

____

☐ ADDITIONAL PAGE(S) ATTACHED

I HEREBY CERTIFY THAT ALL INFORMATION SUBMITTED IN THIS AND ALL ATTACHED DOCUMENTS IS COMPLETE AND ACCURATE AND THAT ALL KNOWN OR SUSPECTED HAZARDS HAVE BEEN DISCLOSED. I FURTHER DECLARE THAT THE SAMPLE I HAVE SUBMITTED FOR ANALYSIS IS REPRESENTATIVE OF THE MATERIAL WHICH I WILL SHIP IF THE SAMPLE IS ACCEPTED.

AUTHORIZED SIGNATURE ____________ TITLE ____________ DATE ____________

LABORATORY	MANUFACTURING	COMPLIANCE

SECTION 4.0

SAMPLING METHOD

4.0 SAMPLING METHOD

The sampling methods outlined below will be followed each time PRC personnel collect samples at the generator's site or from drums or tank trucks brought to PRC. If the generator chooses to collect the samples on his own then he must certify that he has collected a representative sample by following the procedures below or equivalent procedures approved in advance by PRC's laboratory manager. Samples will be collected in accordance with procedures described in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA, SW-846, 1986.

Sampling procedures will be implemented after an initial inspection to determine if the waste matches the description on the Uniform Hazardous Waste Manifest. The color and odor will be checked to determine if the material resembles the identification on the manifest.

4.1 SAMPLING PROCEDURES

The proper sampling procedure is necessary to obtain a representative sample. The basic strategy for profiling of these wastes is the compositing of individual grab samples. Grab samples may be used for screening. Table 4-1 identifies the minimum number of samples to be taken from various sample units.

TABLE 4-1

MINIMUM NUMBER OF SAMPLES

Tank	Three
Drum (coliwasa)	One
Drum (weighted bottle)	Two
Single Tank Truck	
One sample port (coliwasa)	One
One sample port (bottle)	Two
Two sample ports (coliwasa)	Two (composited)
Two sample ports (bottle)	Four (composited)
Multiple sample ports (coliwasa)	Two (composited)
Multiple sample ports (bottle)	Four (composited)

Double Tank Truck without compartments
Each tank will be sampled the same as a single tank truck.

Double Tank Truck with compartments
Compartments contain different waste streams - each compartment sampled the same as a single tank truck.
Compartments contain same waste stream - one compartment sampled the same as a single tank truck.

Pit	Two
Bin	Four
Bag	One

Sampling devices, which are used to sample liquid material, such as coliwasas or weighted bottle samplers, are placed in buckets to prevent any waste from dripping off the device and into the environment.

4.1.1 Aboveground Tank

Tanks are used for accumulating similar material from different batches over a period of time. PRC's objectives in sampling a tank are to determine the layer breaks and identify whether the material exceeds any of the regulatory limits for used oil (hologenated solvents, PCBs, flash point, certain metals). A tank can be characterized if there is sufficient information on the wastes that have been stored in it. The purpose of sampling would then be for marketing reasons. However, if the information on the material is insufficient, a representative sample has to be analyzed. At a minimum, the tank is divided into three equal layers and one sample is taken from each layer. The samples are then composited in the lab prior to analysis. The sampler takes more samples if he has reason to suspect that the three samples will not yield a representative sample. In this situation, the sampler may take samples at graduated intervals of 2 or 3 feet.

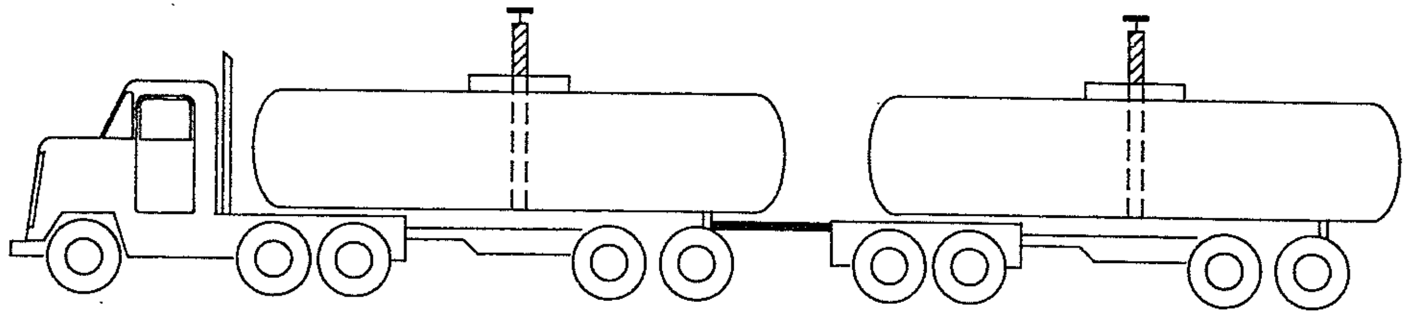
Samples will be taken through the hatch on the top of the tank using a coliwasa or weighted bottle sampler, depending on the depth of the tank and the solids content of the material. Section 4.2 describes sampling equipment.

4.1.2 Tank Truck

Samples will be taken using a coliwasa as soon as possible after the truck stops to avoid excessive separation of material. Samples are collected through the sample hatch on top of the tank or, if safety allows, the manhole. If a load includes more than one tank, then each tank compartment will be sampled separately. If a tank has more than one sample port, a sample will be taken from at least two of the sample ports and composited in the laboratory.

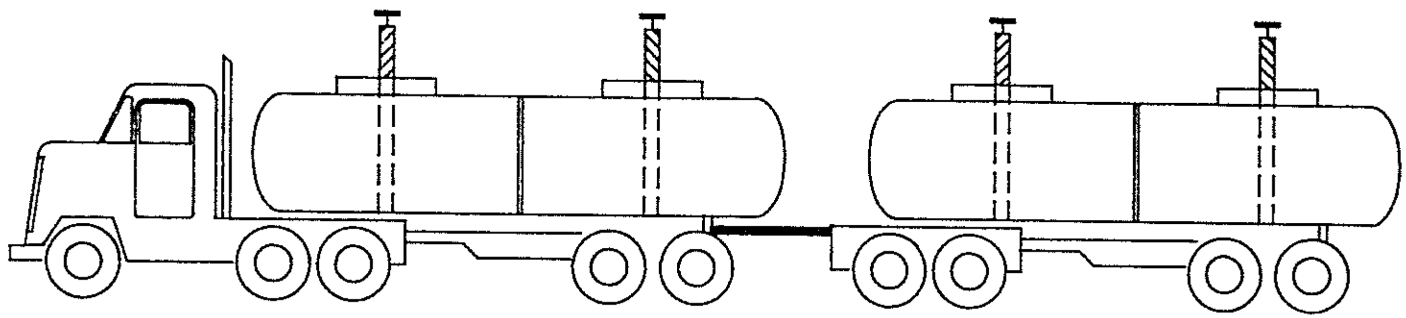
A weighted bottle will be used if the material cannot be sampled using a coliwasa due to solids content. If a weighted bottle is used, then at least two samples will be taken and composited in the laboratory.

Figures 4-1 and 4-2 identify sampling points for tank trucks. Diagrams of sampling equipment as well as procedures for their use are presented in Section 4.2.



DOUBLE TANK TRUCK WITHOUT COMPARTMENTS

1. SAMPLE FROM EACH TANK



DOUBLE TANK TRUCK WITH DUAL COMPARTMENTS

1. COMPARTMENTS CONTAIN DIFFERENT WASTE STREAM: 1 SAMPLE FROM EACH COMPARTMENT

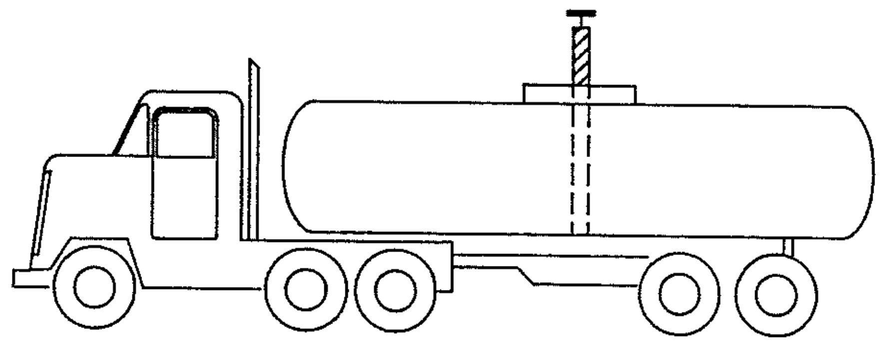
2. COMPARTMENTS CONTAIN SAME WASTE STREAM: 1 SAMPLE FROM RANDOMLY SELECTED COMPARTMENT

ENG	PCR
CHK BY	
APP BY	
DRAWN	KYM
DATE	4/23/91
SCALE	NONE
CAD NO	14970017
FILE NO	P1497-05

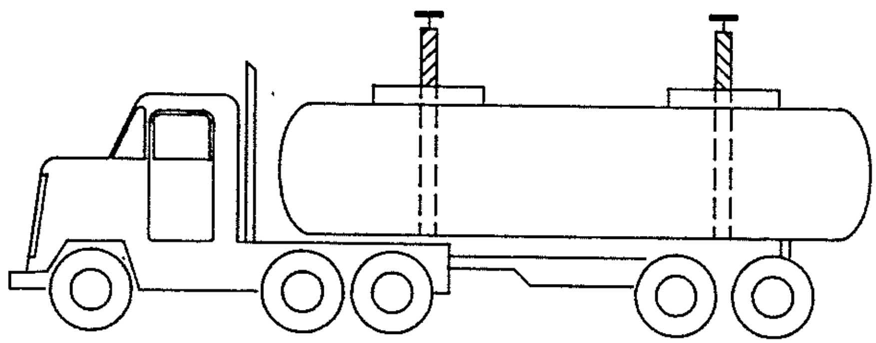
MITTELHAUSER Corporation

DOUBLE TANK TRUCK DRAWING
SAMPLE LOCATIONS
PETROLEUM RECYCLING CORPORATION
SIGNAL HILL FACILITY

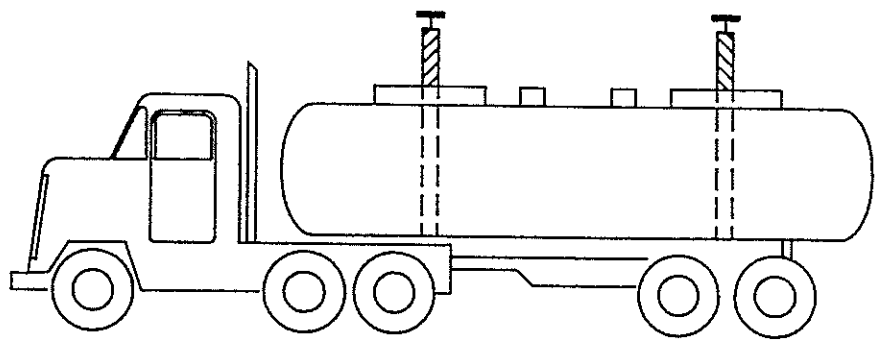
DWG NO. FIGURE 4-2 REV A



SINGLE OPENING
COLIWASA — 1 SAMPLE
W. BOTTLE — 2 SAMPLES COMPOSITED



FRONT AND BACK OPENING
2 SAMPLES COMPOSITED



MULTIPLE OPENINGS
2 SAMPLES COMPOSITED

DWG	PCR
CHK BY	
APP BY	
DRAWN	KYM
DATE	4/23/91
SCALE	NONE
CAD NO	14970016
FILE NO	P1497-05

MITTELHAUSER corporation

SINGLE TANK TRUCK DRAWING
SAMPLE LOCATIONS
PETROLEUM RECYCLING CORPORATION
SIGNAL HILL FACILITY

DWG NO. FIGURE 4-1 REV A

4.1.3 Drum

A coliwasa will be used to collect a column sample. The material captured in the coliwasa is mixed in the laboratory to form one representative sample.

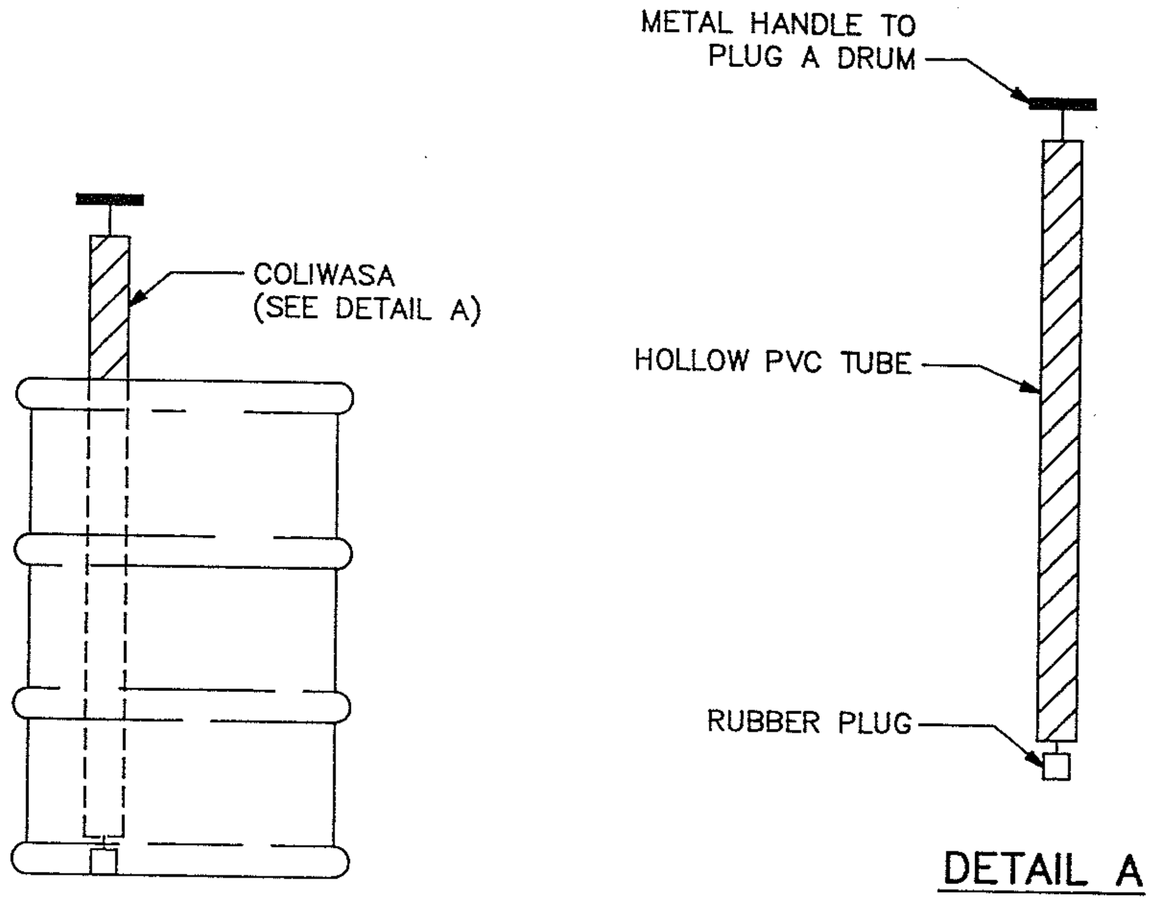
A weighted bottle or scoop device will be used if the material is too solidified to use a coliwasa.

Figure 4-3 is a diagram for identifying the typical sample location for a drum. Sampling equipment is described in Section 4.2.

4.1.4 Pit

Occasionally, PRC will sample a pit such as a clarifier or API pit. PRC can accept material from many types of pits, but rarely samples these locations. Normally, the generator pumps the material from the pit, and samples are taken from the tank where the material is stored prior to being transported.

The waste in the pit may be characterized by knowledge, especially if it falls into a RCRA K- or F-waste category. If there is insufficient knowledge of the composition, then a representative sample will be taken and analyzed.



ENG	PCR
CHK BY	
APP BY	
DRAWN	KYM
DATE	4/23/91
SCALE	NONE
CAD NO	14970015
FILE NO	P1497-05

MITTELHAUSER corporation

LOCATION OF DRUM SAMPLING POINT
PETROLEUM RECYCLING CORPORATION
SIGNAL HILL FACILITY

DWG NO. FIGURE 4-3 REV A

The pit will be examined to identify any deviations in color or appearance. If deviations are noted, then at least two samples will be taken specifically from the areas of concern. If the material in the pit appears homogeneous, two samples will be taken, one from each end. These samples will be composited in the lab prior to analysis.

Depending on the consistency of the material in the pit, either a coliwasa, weighted bottle, or a scoop device will be used to collect the samples.

4.1.5 Solids Bins or Bags

Unit WW1 generates dry solid material that is collected in lined roll-off bins or supersacks prior to disposal or incineration offsite. Samples will be collected periodically during the day as the holding bin fills. Approximately four samples will be collected using a trowel or bucket as the material is pumped from the dryer into the bin. Samples will be composited in the laboratory prior to analysis.

The vapor handling system for WW1 generates spent carbon that is bagged prior to transport offsite for carbon regeneration. Grab samples obtained using a scoop will be used to characterize the spent carbon.

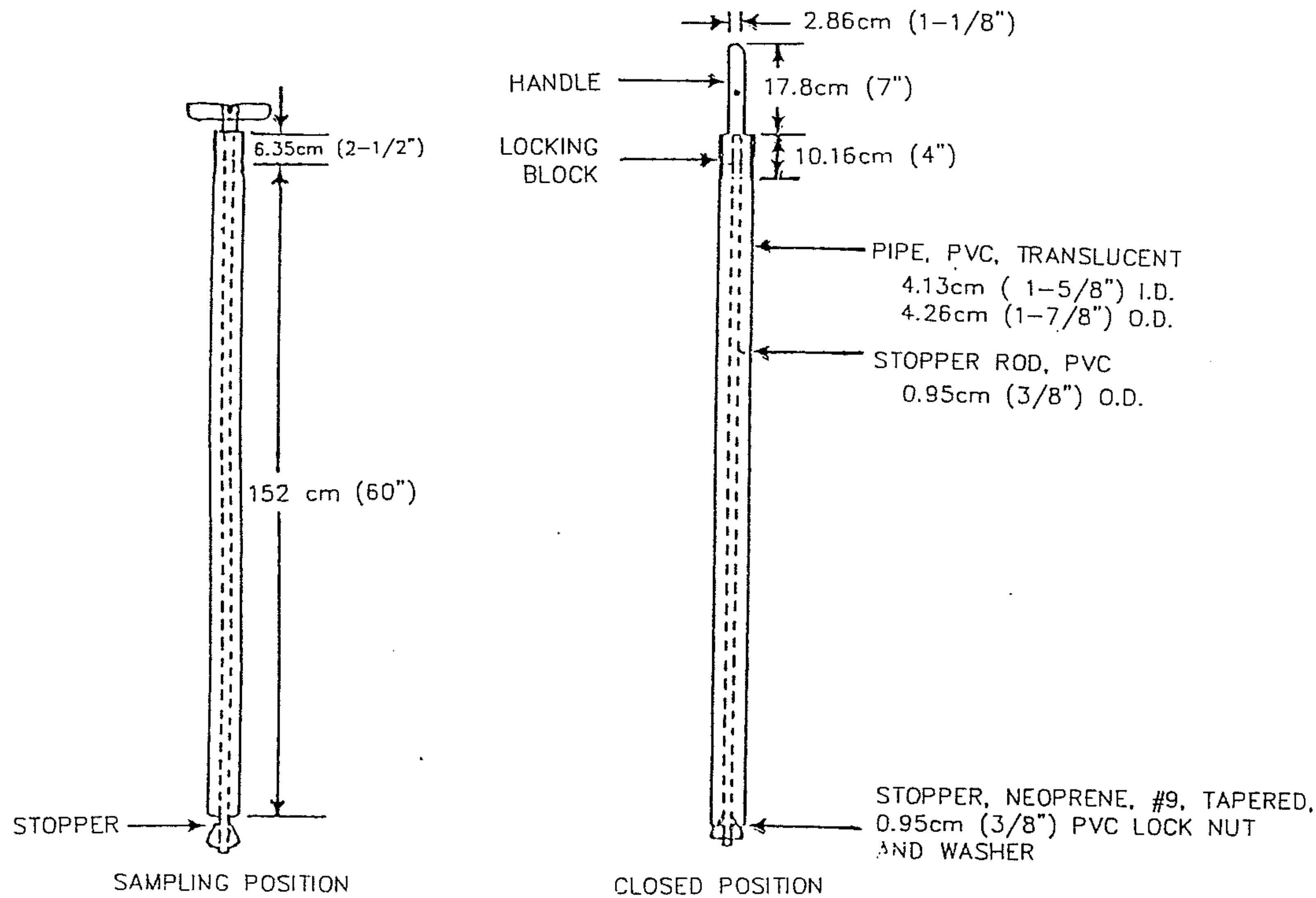
4.2 SAMPLING EQUIPMENT

The proper sampling equipment is essential for obtaining a representative sample. This section describes the design and procedures for use for applicable sampling devices.

4.2.1 Coliwasa

An important liquid sampler for use in sampling solid wastes is the composite liquid waste sampler (coliwasa). This device is both simple and inexpensive, and permits the sampling of both free-flowing liquids and slurries, including multiphase wastes. Coliwasa samples may be collected rapidly, thus minimizing the exposure of the sample collector to the potential hazards of the wastes. In addition, the coliwasa is simple to fabricate and inexpensive enough that contaminated parts may be discarded after a single use if the parts cannot be easily cleaned.

The recommended model of the coliwasa for waste sampling is shown in Figure 4-4. The coliwasa is slowly lowered into the barrel, or other container, holding the waste to be sampled. At the end of the sampler's travel, approximately 1.5 meters, the handle is pulled away from the sampler and rotated to lock the



DSGN	PCR
CHK BY	
APP BY	
DRAWN	KYM
DATE	6/17/91
SCALE	NONE
CAD NO	14970019
BILL NO	P1497-05

MITTELHAUSER corporation

COMPOSITE LIQUID WASTE
SAMPLER (COLIWASA)
SIGNAL HILL FACILITY

DWG NO. FIGURE 4-4

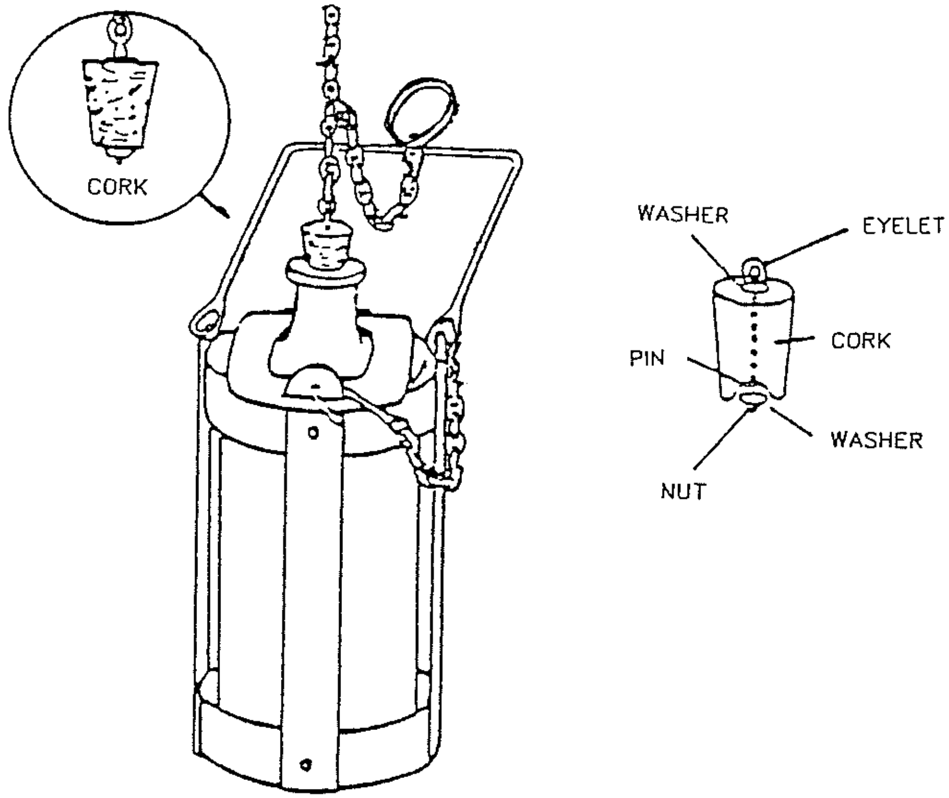
REV A

stopper into the closed position. The liquid sample is then removed from the waste container and transferred to the sample container. This process is repeated until the requisite sample quantity has been collected. The primary limitation on the use of a coliwasa is that the sample depth cannot exceed 1.5 meters.

4.2.2 Weighted Bottle Sampler

The weighted bottle sampler usually consists of a glass bottle, a weighted sinker, a bottle stopper, and a line which is used to raise and lower the bottle during sampling as well as to open the bottle at the appropriate sampling depth.

An example of a weighted bottle sampler is shown in Figure 4-5. This sampler is used to sample liquids contained in storage tanks, wells, sumps, or other containers that cannot be adequately sampled with other liquid sampling devices. The sampler is lowered to the appropriate depth, uncapped, and withdrawn. When removed from the waste, the bottle may be capped, rinsed, and used as the sample storage container. The sampler cannot be used to collect liquids that are incompatible with or chemically react with the weighted sinker or the control lines.



1 QUART WEIGHTED BOTTLE CATCHER
(CAN BE FABRICATED TO FIT ANY SIZE BOTTLE)

DWN	PCR
CHK BY	
APP BY	
DRAWN	KYM
DATE	6/17/91
SCALE	NONE
CAD NO	14970020
FILE NO	P1497-05

MITTELHAUSER Corporation

WEIGHTED BOTTLE SAMPLER
SIGNAL HILL FACILITY

DWG NO. FIGURE 4-5

REV A

4.3 SAMPLE CONTAINERS

Samples of hazardous waste are collected using the sampling equipment previously described and placed in sample containers. The samples are properly recorded, labelled, and sealed as described in Section 4.4. Samples are refrigerated in the laboratory prior to analysis. Samples are archived in the sample storage shed for 30 days after they are analyzed. After 30 days the samples are emptied into the onsite tank used for collection of household oil.

4.4 RECORD KEEPING

Accurate record keeping and chain of custody are essential for proper hazardous waste management. Samples must be traceable from the time they are collected until they are analyzed. Analytical results from each particular sample must be readily available. In addition, if the analyses are ever used for litigation, proper chain of custody procedures must be documented.

4.4.1 <u>Sample Labels and Seals</u>

Samples labels help prevent misidentification of samples. Samples are assigned a unique sample number. Once a waste has been prequalified, then subsequent loads of that waste are identified by using the prequalification number as part of the generator information.

All samples collected by PRC personnel are identified with a label which contains the following information:

- Sample number
- Name of collector
- Date and time of collection
- Sample depth (if applicable)
- Place of collection
- Generator

Attachment 4-1 presents a copy of a sample label.

If a generator collects the sample, then PRC provides him with a label to be completed and attached to the sample for transport to PRC.

Samples collected by PRC personnel and delivered to the PRC laboratory by the sampler do not require seals as long as the sample is under the sampler's direct custody until delivery to the laboratory. However, samples provided by the generator or released from the PRC sampler's custody prior to receipt at the laboratory must be sealed at the time of collection. Sample seals contain the following information:

- Sample number (same as on label)
- Name of collector
- Date and time of collection
- Place of collection

4.4.2 Field Logbook

All information pertinent to sampling is recorded in a bound logbook with consecutively numbered pages. Each sampler who travels to the generator's site has his own logbook. Samples that are taken at the plant are recorded in a logbook kept in the laboratory. The logbook records the following information:

- Date and time
- Sample number
- Sampling location
- Sampling point
- Field contact or driver
- Generator
- Process producing waste
- Waste type
- Number and volume of sample
- Purpose of sampling
- Initials of sampler

4.4.3 Receipt and Logging of Samples by Laboratory

Receipt of samples is noted in the laboratory sample logbook. A sample number is assigned as part of the log-in procedure.

4.4.4 Chain of Custody Records

An essential part of any sampling/analytical scheme is ensuring the integrity of the sample from collection to data reporting. The possession and handling of samples should be traceable from the time of collection through analysis and final disposition. This documentation of the history of the sample is referred to as chain of custody.

Chain of custody is necessary if there is any possibility that the analytical data or conclusions based upon analytical data will be used in litigation.

A sample is considered to be under a person's custody if it is: (1) in a person's physical possession, (2) in view of the person after taking possession, and (3) secured by that person in an area that is restricted to authorized personnel only.

Attachment 4-1 contains a copy of a chain of custody form.

4.4.5 Sample Analysis Request Sheet

A prequalification survey is completed either by the generator or from information collected from the generator as part of the profiling process for all waste streams. The prequalification survey of the waste identifies generator information such as EPA ID number and the waste generating process; physical characteristics such as color, odor, layers, etc.; chemical composition; metals concentration; shipping information; hazardous characteristics such as reactivity or radioactivity; special handling information; and certification of representative sampling and sampling procedure.

A copy of the prequalification survey is presented in Attachment 4-1.

4.5 SAMPLES TAKEN BY NONFACILITY PERSONNEL

If nonfacility personnel perform sampling activities, the sampler must certify that he/she has collected a representative sample. As part of the certification, the sampling procedures used must be identified on the prequalification survey.

4.6 DECONTAMINATION PROCEDURES

The sampling equipment will be cleaned at the beginning of the sampling process, between each sample, and at the end of the sampling process to avoid bringing contaminants away from the site. Cleaning will include rinsing of equipment with a petroleum distillate such as diesel.

Contaminated clothing and personal equipment will either be sent to a laundry facility or disposed of.

4.7 HEALTH AND SAFETY

All PRC personnel who perform sampling of hazardous wastes participate in an appropriate health and safety program. Any employee who performs field work of this type is medically monitored as required under 29 CFR 1910.120(b)(5). Prior to being authorized to collect samples of hazardous waste, personnel are instructed on the common routes of exposure to chemicals (i.e., inhalation, contact, ingestion) and in the proper use of protective clothing and respiratory equipment in accordance with 29 CFR 1910.120(b)(4).

The following items of personal protective equipment are worn when applicable by all field personnel when collecting samples:

- Hard hat
- Gloves
- Safety boots
- Respirator

A first aid kit is carried onsite as part of the sampling equipment. All PRC personnel are trained in proper first aid procedures at the time of employment and through periodic refresher training.

ATTACHMENT 4-1

SAMPLING DOCUMENTATION

- Sample Label
- Chain of Custody Form
- Prequalification Survey

MITTELHAUSER corporation

PRC

SAMPLE NO.______________

SAMPLE DATE______________

SAMPLE TIME______________

NAME OF COLLECTOR______________________________

SAMPLE LOCATION______________________________

SAMPLE DEPTH (IF APPLICABLE)____________________

GENERATOR____________________________________

PROFILE NO.__________________________________

[1185AL]

PP RESOURCES RECOVERY
2651 Walnut Avenue
Signal Hill, CA 90806
Office: 213-595-7431
Plant: 213-595-8503

CHAIN OF CUSTODY RECORD

PROJECT NUMBER:

PROJECT NAME:

SAMPLED BY: (PRINTED AND SIGNATURE)

SAMPLE NUMBER	DATE	TIME	TYPE	SAMPLE LOCATION	NUMBER OF CONTAINERS	ANALYSIS(ES):						PRESERVATIVE	REMARKS

RELINQUISHED BY: (SIGNATURE)	DATE	TIME	RECEIVED BY: (SIGNATURE)	TOTAL NO. OF SAMPLES (THIS SHIPMENT) / TOTAL NO. OF CONTAINERS (THIS SHIPMENT)	LABORATORY:
RELINQUISHED BY: (SIGNATURE)	DATE	TIME	RECEIVED BY: (SIGNATURE)	LABORATORY CONTACT:	LABORATORY PHONE NUMBER: ( )
RELINQUISHED BY: (SIGNATURE)	DATE	TIME	RECEIVED FOR LABORATORY BY: (SIGNATURE)	SAMPLE ANALYSIS REQUEST SHEET ATTACHED: ( )YES ( )NO	

DISTRIBUTION: WHITE, PRC
CANARY, LABORATORY
PINK, CLIENT
GOLD, PROJECT FILE

REMARKS:

PRE-QUALIFICATION SURVEY

PETROLEUM RECYCLING CORP.

OFFICE: 2651 Walnut Avenue, Signal Hill, California 90806 (213) 595-7431
PLANT: 1835 East 29th Street, Signal Hill, California 90806 (213) 595-6597

PQS #

APPROVAL #

A GENERATOR INFORMATION

NAME: ______

ADDRESS: ______

USEPA ID# ______

REPRESENTATIVE INFORMATION:

NAME: ______

ADDRESS: ______

PHONE #'S ______

TECHNICAL CONTACT: ______ TITLE: ______ PHONE: ______

PRC SALES REP: ______

NAME OF WASTE ______

PROCESS GENERATING WASTE ______

B PHYSICAL CHARACTERISTICS OF WASTE (SHOW ANTICIPATED RANGES)

COLOR

ODOR ☐ NONE ☐ MILD ☐ STRONG ______

PHYSICAL STATE @ 70°F ☐ SOLID ☐ SEMI-SOLID ☐ LIQUID

LAYERS ☐ MULTILAYERED ☐ BI-LAYERED ☐ SINGLE PHASED

FREE LIQUIDS ☐ YES VOLUME % ______

BTU VALUE BTU/LB. ______

pH ANTICIPATED RANGE ☐ ≤ 2 ☐ > 2 - 4 ☐ 4.1 - 10 ☐ 10.1 - 12.4 ☐ $\geq$ 12.5 ☐ EXACT

DENSITY OR API GRAVITY ______ ☐ LIQUID lb/gal ☐ SOLID lbs/ft

FLASH POINT ☐ < 70°F ☐ 70°F - 100°F ☐ 101°F - 139°F ☐ 140°F - 200°F ☐ > 200°F ☐ NO FLASH ☐ EXACT ______ ☐ OPEN ☐ CLOSED CUP

C CHEMICAL COMPOSITION (TOTALS MUST ADD TO 100%)

	%
	%
	%
	%
	%
	%
	%
	%
	%
	%
	%

D METALS ☐ TOTAL (PPM) ☐ EPA EXTRACTION PROCEDURE (mg/L)

ARSENIC		SELENIUM (Se)	
BARIUM (Ba)		SILVER (Ag)	
CADMIUM (Cd)		COPPER (Cu)	
CHROMIUM		NICKEL (Ni)	
MERCURY (Hg)		ZINC (Zn)	
LEAD (Pb)		THALLIUM (Tl)	

E OTHER COMPONENTS - TOTAL (PPM)

CYANIDES ______ PCB S ______

SULFIDES ______ PHENOLICS ______

F SHIPPING INFORMATION

PROPER SHIPPING NAME: ______

HAZARD CLASS ______ I.D.NO. ______

METHOD OF SHIPMENT ☐ BULK LIQUID ☐ DRUM (TYPE/SIZE) ______

ANTICIPATED VOLUME ______ GALS ______ OTHER ______

PER ☐ ONE TIME ☐ WEEK ☐ MONTH ☐ QUARTER ☐ YEAR ______ ☐ ______

G HAZARDOUS CHARACTERISTICS

REACTIVITY ☐ NONE ☐ PYROPHORIC ☐ SHOCK SENSITIVE ☐ EXPLOSIVE ☐ WATER REACTIVE ☐ OTHER ______

OTHER HAZARDOUS CHARACTERISTICS

☐ NONE ☐ RADIOACTIVE ☐ ETIOLOGICAL ☐ PESTICIDE MANUFACTURING WASTE ☐ OTHER ______

USEPA HAZARDOUS WASTE? ☐ YES ☐ NO

USEPA HAZARDOUS CODE(S) ______

STATE HAZARDOUS WASTE? ☐ YES ☐ NO

STATE CODE(S) ______

H SPECIAL HANDLING INFORMATION ______

☐ ADDITIONAL PAGE(S) ATTACHED

I HEREBY CERTIFY THAT ALL INFORMATION SUBMITTED IN THIS AND ALL ATTACHED DOCUMENTS IS COMPLETE AND ACCURATE AND THAT ALL KNOWN OR SUSPECTED HAZARDS HAVE BEEN DISCLOSED. I FURTHER DECLARE THAT THE SAMPLE I HAVE SUBMITTED FOR ANALYSIS IS REPRESENTATIVE OF THE MATERIAL WHICH I WILL SHIP IF THE SAMPLE IS ACCEPTED

AUTHORIZED SIGNATURE ______ TITLE ______ DATE ______

LABORATORY	MANUFACTURING	COMPLIANCE

SECTION 5.0

ANALYTICAL PROCEDURES

5.0 ANALYTICAL PROCEDURES

Representative samples will be evaluated using the analytical methods outlined in this section.

5.1 RATIONALE FOR SELECTION OF ANALYTICAL PARAMETERS

The primary criteria for selection of analytical parameters are the requirements of 40 CFR 264.13 and 22 CCR 67102 that specify that before the treatment, storage, or disposal of any hazardous waste, a representative sample must be analyzed for all parameters that provide information necessary to treat, store, or dispose of the waste. In addition, parameters were selected depending upon the waste's expected disposition, namely recycling or transporting to a disposal or incineration facility.

Selection of analytical parameters was based on the following factors:

- The oil-water wastes contain fuel and lubricating oils that may contain trace metals.
- The specific processes that created the waste, such as API separation or tank cleaning.
- The intended market use of the recycled material.
- The facility's industrial wastewater discharge permit that identifies effluent limitations on wastewater.

- Constituents identified at 40 CFR 261 Appendix VII as the basis for listing K- and F-stream wastes.
- Used oil specifications found at 40 CFR 266 and Article 13, Chapter 6.5, of the California Health & Safety Code, Section 25250.
- Properties of wastes that could cause a system upset.

5.2 PARAMETERS

Each waste stream has two sets of analytical parameters associated with it - prequalification profiling and screening. Once a particular waste from a specific generator has been approved (prequalified), then future shipments of that particular waste are screened for particular constituents. If prequalification analyses reveal some constituents to be borderline, those constituents are analyzed each time that waste stream is received at PRC. Profiling analytical results are computerized. Each time a shipment of a waste arrives the profile results can be accessed so that borderline constituents can be easily identified.

The following parameters are measured during the initial profiling of all wastes. If a generator chooses to provide his own profile, then it must include information on these parameters.

- Flash point
- BS&W
- Total Organic Halogens (water streams)
- Total Halogens (oil streams)
- PCBs
- pH
- Color
- Odor
- API Gravity/Specific Gravity
- Total sulfides

In addition, Title 22 metals are measured for oily water streams (streams containing greater than 20 percent water). Metals are measured for other waste streams when necessary to determine proper treatment. Other parameters are measured when based on origin of the waste or the generating process. For example, cyanides will be measured for wastewater streams generated through plating operations.

Color, odor, BS&W, and API gravity/specific gravity are checked as part of the screening on every load. In addition, total organic halogens or total halogens are analyzed for every shipment unless it is designated as a solvent-containing waste on the manifest. Sulfides are analyzed for those wastes which exhibited a total sulfide content of 250 ppm or greater during profiling. Other parameters will be analyzed on a random basis or if the prequalification profile indicates borderline concentrations. Table 5-1 identifies verification methods evaluated during screening.

TABLE 5-1

VERIFICATION METHODS

Waste Type	Verification Analysis
Used Oil and Petroleum Refining Listed Hazardous wastes	total halogens/chlorine BS&W API/specific gravity color odor
Oily Water	total organic halogens one random heavy metal BS&W API/specific gravity color odor
Alternative Fuel	total halogens Btu content BS&W API/specific gravity color odor
Antifreeze	total organic halogens one random heavy metal BS&W API/specific gravity color odor

Hazardous wastes generated by PRC's treatment processes are limited to solids and water streams from WW1 (centrifuge/dryer/filter press) and WWT (wastewater treatment system). Tank bottoms from oil receiving/treatment/storage tanks are processed through WW1. In addition, two wastes may be generated by the vapor handling systems either in place or to be installed at the facility. The vapor handling system on WW1 generates spent carbon (which may or may not be a hazardous waste due to benzene concentration) from the four carbon canisters. The carbon canisters are emptied into bags prior to transport of the carbon to a carbon regeneration facility. The proposed vapor handling system for the tanks and loading/unloading racks at the facility will generate spent caustic scrubber that will be drummed prior to being transported to a recycling facility.

Any wastes resulting from the storage of materials for use as alternative fuel are transported to a permitted facility that can handle the waste material.

Wastes being sent offsite for treatment or disposal may be subject to federal Land Disposal Restrictions if they are federal hazardous wastes. See Section 6.0 for procedures to comply with these regulations.

Wastes being sent offsite which are identified as containing no free liquids (i.e., dry solids in roll-off bins) must pass the paint filter test in order to demonstrate that no free liquids are present. Free liquids are not present when a 100 ml representative sample of the waste can be completely retained in a standard 400 micron conical paint filter for 5 minutes without loss of any portion of the waste from the bottom of the filter.

5.3 TEST METHODS

Representative samples will be analyzed using the analytical methods identified on Table 5-2.

All analytical procedures for pH, flash point, and other tests deemed appropriate are conducted in accordance with the methods identified in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, 3rd Edition, U.S. EPA, 1986; "Methods for Chemical Analysis of Water and Waste," EPA-600/4-79-020, U.S. EPA, 1979; applicable ASTM standards and "Standard Methods for the Examination of Water and Wastewater," (17th Edition), American Public Health Administration (1989). Detection limits for specific analytical methods are also identified.

TABLE 5-2 cont'd

ANALYTICAL METHODS

Parameter	Method	Reference	Detection Limit (ppm)
pH	9040	SW-846	N/A
Sulfides	Test kit for total sulfides		1.0
Cyanides	9010 or test kit	SW-846	0.5
Color	None		
Odor	None		
API gravity	D88-79	A.S.T.M.	N/A
Oil & grease	413.2	EPA 600	1.0 ppb

All methods in use at PRC except 9076 have been approved by the EPA. Method 9076 is a draft EPA method for analysis of total halogens. Attachment 5-1 contains a description of this draft method. Test kits such as those for total organic halogens, sulfides, or cyanides may also be used.

5.4 QA/QC PROCEDURES

QA/QC procedures will be conducted by following the specific procedures associated with each analytical method and included in the descriptions of the methods. The laboratory will maintain a quality control notebook that will keep track of each time the method is run, when duplicates are due, and the results of those duplicates. Any discrepancies will be reported to the laboratory manager to investigate.

Each item of analytical equipment has its own maintenance file. Information maintained in this file includes results from periodic QA/QC testing, scheduled and unscheduled repairs, as well as any difficulties or problems.

5.5 RECORD KEEPING

As samples are analyzed, the results are noted on laboratory logbook sheets. These results are entered in the computer once all the analyses on a particular sample are complete.

5.5.1 Numbering and Documenting Path of Samples

Samples are assigned sample numbers when they are received and logged in. Analytical results are computerized by sample number, generator, and shipper. Results from screening analyses are also computerized by sample number, generator, and shipper. As a treatment facility, PRC maintains copies of all analyses for the lifetime of the facility.

5.5.2 Destiny of Remaining Sample After Analysis

Samples are stored for 30 days after analyses are completed. After 30 days, the samples are emptied into an appropriate tank used to collect waste. This tank is emptied periodically after being sampled and analyzed. The oil is properly routed to either the used oil recycling program or the alternative fuel program, whichever is appropriate.

5.5.3 Documentation Filed by Manager

Copies of the following are kept on file in the PRC laboratory: prequalification survey, test results, chain of custody forms, recertification forms, certification of representative sample forms (if applicable), and analyses of any screening tests.

5.6 WASTES ANALYZED OUTSIDE THE FACILITY

The generator of a waste steam must sign a certification of representative sample for any sample not collected by PRC personnel, regardless of whether the sample is analyzed at PRC or an outside laboratory. This certification is located on the prequalification survey and includes information on the sampling procedures used to obtain the sample (see Attachment 4-1). A copy of any analytical results from an outside laboratory indicating the method performed, detection limit, and result must be attached to the prequalification survey.

Outside laboratories performing analytical work for profiling of wastes should be certified by the DHS. Verification of a laboratory's certification can be made by calling the DHS Laboratory Section in Berkeley or referring to published lists of certified laboratories.

ATTACHMENT 5-1

EPA DRAFT METHOD 9076

TOTAL CHLORINE IN OIL

HYDROGEN SULFIDE TEST KIT
Range 0-5 mg/L as Hydrogen Sulfide
Model HS-7
Cat. No. 2239-00

HACH

To ensure accurate results read carefully before proceeding.

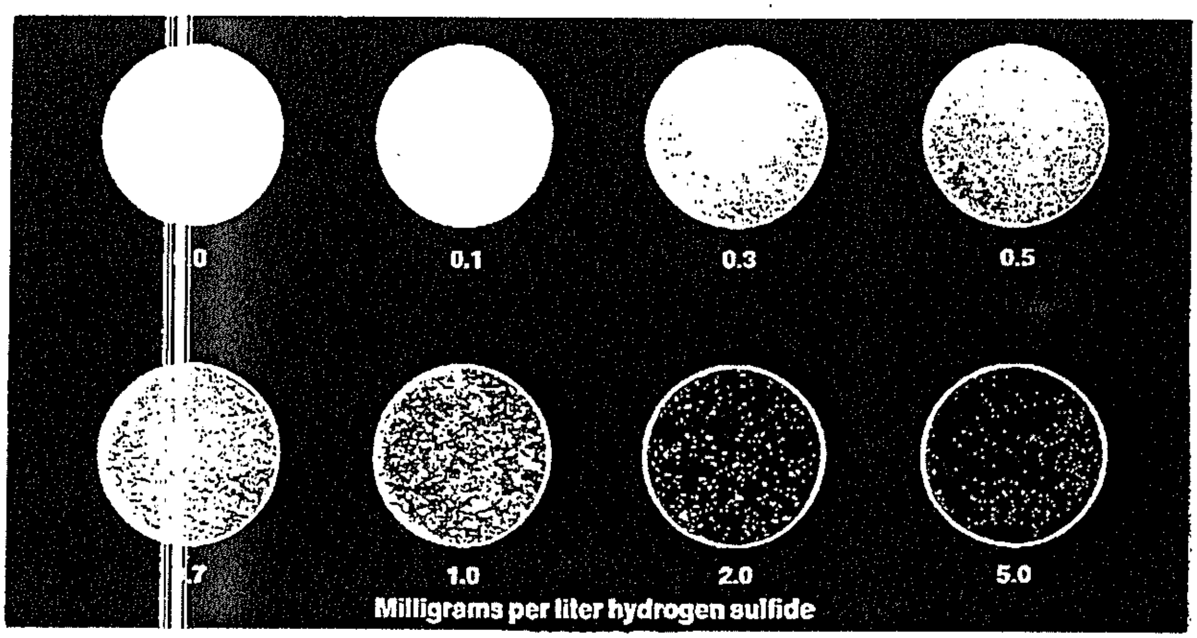
The test for hydrogen sulfide should be performed on water that has been freshly pumped. If the water has been aerated or allowed to stand before testing, much, if not all, of the hydrogen sulfide will be lost through aeration and oxidation.

Test Instructions

1. Fill the sample vial to the 25-mL mark with the water to be tested for hydrogen sulfide.
2. Place a circle of Hydrogen Sulfide Test Paper inside the cap of the sample vial.
3. Add an Alka Seltzer® tablet to the water sample and **immediately** snap the cap containing the test paper onto the vial.
4. After the tablet has dissolved, remove the test paper and compare the color of the test paper with the color chart.

WARNING: The chemicals in this kit may be hazardous to the health and safety of the user if inappropriately handled. Please read all warnings before performing the test and use appropriate safety equipment.

HACH COMPANY, P.O. BOX 389. LOVELAND, COLORADO 80539
TELEPHONE: WITHIN U.S. 800-227-4224, OUTSIDE U.S. 303-669-3050, TWX 910-930-9038



ROSEMOUNT ANALYTICAL INC.

DOHRMANN DIVISION
Technical Reprint

TR-024

US EPA METHOD 9076

TEST METHOD FOR TOTAL CHLORINE IN NEW AND USED PETROLEUM PRODUCTS BY OXIDATIVE COMBUSTION AND MICROCOULOMETRY

EPA Method 9076 is an officially accepted Method for the Determination Total Chlorine in Used Oil.

Reprinted by Rosemount Analytical Inc., Dohrmann Division, 3240 Scott Blvd. Santa Clara, CA 95054, with editoral corrections

TEST METHOD FOR TOTAL CHLORINE IN NEW AND USED PETROLEUM PRODUCTS BY OXIDATIVE COMBUSTION AND MICROCOULOMETRY

1.0 SCOPE AND APPLICATION

1.1 This test method covers the determination of total chlorine in new and used oils, fuels and related materials, including crankcase, hydraulic, diesel, lubricating and fuel oils, and kerosene by oxidative combustion and microcoulometry. The chlorine content of petroleum products is often required prior to their use as a fuel.

1.2 The applicable range of this method is from 10 to 10,000 ug/g chlorine.

2.0 SUMMARY OF METHOD

2.1 The sample is placed in a quartz boat at the inlet of a high temperature quartz combustion tube. An inert carrier gas such as argon, carbon dioxide, or nitrogen sweeps across the inlet while oxygen flows into the center of the combustion tube. The boat and sample are advanced into a vaporization zone of approximately 300°C to volatilize the light ends. Then the boat is advanced to the center of the combustion tube, which is at 1,000°C. The oxygen is diverted to pass directly over the sample to oxidize any remaining refractory material. All during this complete combustion cycle, the chlorine is converted to chloride and oxychlorides, which then flow into an attached titration cell where they quantitatively react with silver ions. The silver ions thus consumed are coulometrically replaced. The total current required to replace the silver ions is a measure of the chlorine present in the injected samples.

2.2 The reaction occurring in the titration cell as chloride enters is:

$$Cl^- + Ag^+ \longrightarrow AgCl\ (s)$$

The silver ion consumed in the above reaction is generated coulometrically thus:

$$Ag^o \longrightarrow Ag^+ + e^-$$

2.3 These microequivalents of silver are equal to the number of microequivalents of titratable sample ion entering the titration cell.

3.0 INTERFERENCES

3.1 Other titratable halides will also give a positive response. These titratable halides include HBr and HI (HOBr + HOI do not precipitate silver). Because these oxyhalides do not react in the titration cell, approximately 50% microequivalent response is detected from bromine and iodine.

3.2 Fluorine as fluoride does not precipitate silver, so it is not an interferent, nor is it detected.

3.3 This test method is applicable in the presence of total sulfur concentrations of up to 10,000 times the chlorine level.

4.0 APPARATUS AND MATERIALS

4.1 Combustion furnace. The sample should be oxidized in an electric furnace capable of maintaining a temperature of 1,000°C to oxidize the organic matrix.

4.2 Combustion tube. Fabricated from quartz and constructed so that a sample, which is vaporized completely in the inlet section, is swept into the oxidation zone by an inert gas where it mixes with oxygen and is burned. The inlet end of the tube connects to a boat insertion device where the sample can be placed on a quartz boat by syringe, micropipet, or by being weighed externally. Two gas ports are provided, one for an inert gas to flow across the boat and one for oxygen to enter the combustion tube.

4.3 Microcoulometer, having variable gain and bias control, and capable of measuring the potential of the sensing-reference electrode pair, and comparing this potential with a bias potential, and applying the amplified difference to the working-auxiliary electrode pair so as to generate a titrant. The microcoulometer output signal shall be proportional to the generating current. The microcoulometer may have a digital meter and circuitry to convert this output signal directly to nanograms or micrograms of chlorine or micrograms per gram chlorine.

4.4 Titration cell. Two different configurations have been applied to coulometrically titrate chlorine for this method.

4.4.1 Type I uses a sensor-reference pair of electrodes to detect changes in silver ion concentration and a generator anode-cathode pair of electrodes to maintain constant silver ion concentration, and an inlet for a gaseous sample from the pyrolysis tube. The sensor, reference, and anode electrodes are silver electrodes. The cathode electrode is a platinum wire. The reference electrode resides in a saturated silver acetate half-cell. The electrolyte contains 70% acetic acid in water.

4.4.2 Type II uses a sensor-reference pair of electrodes to detect changes in silver ion concentration and a generator anode-cathode pair of electrodes to maintain constant silver ion concentration, an inlet for a gaseous sample that passes through a 95% sulfuric acid dehydrating tube from the pyrolysis tube, and a sealed two-piece titration cell with an exhaust tube to vent fumes to an external exhaust. All electrodes can be removed and replaced independently without reconstructing the cell assembly. The anode electrode is constructed of silver. The cathode electrode is constructed of platinum. The anode is separated from the cathode a 10% KNO_3 agar bridge, and continuity is maintained through an aqueous 10% KNO_3 salt bridge. The sensor electrode is constructed of silver. The reference electrode is a silver/silver chloride ground glass sleeve, double-junction electrode with aqueous 1M KNO_3 in the outer chamber and aqueous 1M KCl in the inner chamber.

4.5 Sampling syringe, a microliter syringe of 10 uL capacity capable of accurately delivering 2 to 5 uL of a viscous sample into the sample boat.

4.6 Micropipet, a positive displacement micropipet capable of accurately delivering 2 to 5 uL of a viscous sample into the sample boat.

4.7 Analytical balance. When used to weigh a sample of 2 to 5 mg onto the boat, the balance shall be accurate to $\pm$ 0.01 mg. When used to determine the density of the sample, typically 8 g per 10 mL, the balance shall be accurate to $\pm$ 0.1 g.

5.0 REAGENTS

5.1 Purity of Reagents. Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[2] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Water. Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of ASTM Specification D1193.

5.3 Acetic acid, CH_3CO_2H. Glacial.

5.4 Isooctane, $(CH_3)_2CHCH_2C(CH_3)_3$.

5.5 Chlorobenzene, C_6H_5Cl.

5.6 Chlorine, standard stock solution - 10,000 ng Cl/uL. Weigh accurately 3.174 g of chlorobenzene into 100 mL volumetric flask. Dilute to the mark with isooctane.

5.7 Chlorine, standard solution. 1,000 ng Cl/uL, pipet 10.0 mL of [10] chlorine stock solution (Step 5.6) into a 100-mL volumetric flask and dilute to volume with isooctane.

5.8 Argon, helium, nitrogen, or carbon dioxide, high-purity grade (HP) used as the carrier gas.

5.9 Oxygen, high-purity grade (HP), used as the reactant gas.

5.10 Gas regulators. Two-stage regulator must be used on the reactant and carrier gas.

5.11 Cell Type I.

5.11.1 Cell electrolyte solution. 70% acetic acid, combine 300 mL water (Step 5.2) with 700 mL acetic acid (Step 5.3) and mix well.

5.11.2 Silver acetate, CH_3CO_2Ag. Powder purified for saturated reference electrode.

[2] "Reagent Chemicals, American Chemical Society Specifications," American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards." by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia".

5.12 Cell Type 2.

5.12.1 Sodium acetate, CH_3CO_2Na.

5.12.2 Potassium Nitrate, KNO_3.

5.12.3 Potassium chloride, KCl.

5.12.4 Sulfuric acid (concentrated), H_2SO_4.

5.12.5 Agar, (jelly strength 450 to 600 g/cm²).

5.12.6 Cell electrolyte solution - 85% acetic acid, combine 150 mL water (Step 5.2) with 1.35 g sodium acetate (Step 5.12.1) and mix well; add 850 mL acetic acid (Step 5.3) and mix well.

5.12.7 Dehydrating solution - Combine 95 mL sulfuric acid (Step 5.12.4) with 5 mL water (Step 5.2) and mix well.

CAUTION: This is an exothermic reaction and may proceed with bumping unless controlled by the addition of sulfuric acid. Slowly add sulfuric acid to water. Do not add water to sulfuric acid.

5.12.8 Potassium nitrate (10%), KNO_3. Add 10 g potassium nitrate (Step 5.12.2) to 100 mL reagent water (Step 5.2) and mix well.

5.12.9 Potassium nitrate (1M), KNO_3. Add 10.11 g potassium nitrate (Step 5.12.2) to 100 mL reagent water (Step 5.2) and mix well.

5.12.10 Potassium chloride (1M), KCl. Add 7.46 g potassium chloride (Step 5.12.3) to 100 mL water (Step 5.2) and mix well.

5.12.11 Agar bridge solution - Mix 0.7 g agar (Section 5.12.5), 2.5 g potassium nitrate (Section 5.12.2), and 25 mL reagent water (Section 5.2) and heat to boiling.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Collect a sample of oil representative of the source being sampled using the appropriate SW-846 sampling method. Because the collected sample will be analyzed for total halogens, it should be kept headspace free and refrigerated prior to preparation and analysis to minimize volatilization losses of organic halogens. Because waste oils may contain toxic and/or carcinogenic substances, appropriate field and laboratory safety procedures should be followed.

6.2 Laboratory subsampling of the sample should b performed on a well-mixed sample of oil.

7.0 PROCEDURES

7.1 Preparation of apparatus

7.1.1 Set up the analyzer as per the equipment manufacturer's instructions.

7.1.2 Typical operating conditions: Type I.

Furnace temperature	1,000°C
Carrier gas flow	43 cm³/mi
Oxygen gas flow	160 cm³/mi
Coulometer	
Bias	250 mV
Gain	25%

7.1.3 Typical operating conditions: Type II.

Furnace temperature	H-1 850° C
	H-2 1,000°C
Carrier gas flow	250 cm³/min
Oxygen gas flow	200 cm³/min
Coulometer	
End point potential (bias)	
Gain G-1	1.5 coulombs/mV
G-2	3.0 coulombs/mV
G-3	3.0 coulombs/mV
ES-1 (range 1)	25 mV
ES-2 (range 2)	30 mV

NOTE: Other conditions may be appropriate. Refer to th instrumentation manual.

7.2 Sample introduction.

7.2.1 Carefully fill a 10-uL syringe with 2 to 5 uL c sample depending on the expected concentration of tota chlorine. Inject the sample through the septum onto th cool boat, being certain to touch the boat with the needl tip to displace the last droplet.

7.2.2 For viscous samples that cannot be draw into the syringe barrel, a positive displacement micropipe may be used. Here, the 2-5 uL of sample is placed on th boat from the micropipet through the opened hatch por The same technique as with the syringe is used to dis place the last droplet into the boat. A tuft of quartz woo in the boat can aid in completely transferring the sampl from the micropipet into the boat.

NOTE: Dilution of samples to reduce viscosity is not recommended due to uncertainty about the solubility of the sample and its chlorihated constitutents. If a positive displacement micropipet in not available, dilution may be attempted to enable injection of viscous samples.

7.3.2 Repeat the measurement of this standard at least three times.

7.3.3 System blank - The blank should be checked daily with isooctane. It is typically less than 1 ug/g

5.12 Cell Type 2.

5.12.1 Sodium acetate, CH_3CO_2Na.

5.12.2 Potassium Nitrate, KNO_3.

5.12.3 Potassium chloride, KCl.

5.12.4 Sulfuric acid (concentrated), H_2SO_4.

5.12.5 Agar, (jelly strength 450 to 600 g/cm^2).

5.12.6 Cell electrolyte solution - 85% acetic acid, combine 150 mL water (Step 5.2) with 1.35 g sodium acetate (Step 5.12.1) and mix well; add 850 mL acetic acid (Step 5.3) and mix well.

5.12.7 Dehydrating solution - Combine 95 mL sulfuric acid (Step 5.12.4) with 5 mL water (Step 5.2) and mix well.

CAUTION: This is an exothermic reaction and may proceed with bumping unless controlled by the addition of sulfuric acid. Slowly add sulfuric acid to water. Do not add water to sulfuric acid.

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6.2 Laboratory subsampling of the sample should be performed on a well-mixed sample of oil.

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Carrier gas flow..........................	43 cm^3/min
Oxygen gas flow.......................	160 cm^3/min
Coulometer	
Bias...	250 mV
Gain..	25%

7.1.3 Typical operating conditions: Type II.

Furnace temperature................	H-1 850° C
	H-2 1,000°C
Carrier gas flow......................	250 cm^3/min
Oxygen gas flow......................	200 cm^3/min
Coulometer	
End point potential (bias)	
Gain G-1...........................	1.5 coulombs/mV
G-2...........................	3.0 coulombs/mV
G-3...........................	3.0 coulombs/mV
ES-1 (range 1)......................	25 mV
ES-2 (range 2)......................	30 mV

NOTE: Other conditions may be appropriate. Refer to the instrumentation manual.

7.2 Sample introduction.

7.2.1 Carefully fill a 10-uL syringe with 2 to 5 uL of sample depending on the expected concentration of total chlorine. Inject the sample through the septum onto the cool boat, being certain to touch the boat with the needle tip to displace the last droplet.

7.2.2 For viscous samples that cannot be drawn into the syringe barrel, a positive displacement micropipet may be used. Here, the 2-5 uL of sample is placed on the boat from the micropipet through the opened hatch port. The same technique as with the syringe is used to displace the last droplet into the boat. A tuft of quartz wool in the boat can aid in completely transferring the sample from the micropipet into the boat.

8.0 QUALITY CONTROL

8.1 Each sample should be analyzed twice. If the results do not agree to within 10% expressed as the relative percent difference of the results, repeat the analysis.

8.2 One sample in ten should be spiked with a chlorinated organic at a level of total chlorine commensurate with the levels being determined. The spike recovery should be reported and should be between 80 and 120% of the expected value. Any sample suspected of containing > 25% water should also be spiked with organic chlorine.

9.0 METHOD PERFORMANCE

These data are based on 66 data points obtained by 10 laboratories who each analyzed four used crankcase oils and three fuel oil blends with crankcase in duplicate.[1] A data point represents one duplicate analysis of a sample. One laboratory and four additional data points were determined to be outliers and are not included in these results.

9.1 Precision. The precision of the method as determined by the statistical examination of interlaboratory test results is as follows:

Repeatability - The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would exceed, in the long run, in the normal and correct operation of the test method the following values only in 1 case in 20 (see Table 1):

$$\text{Repeatability} = 0.137 \sqrt{x}^*$$

*where x is the average of two results in ug/g.

Reproducibility - The difference between two single and independent results obtained by different operators working in different laboratories on identical test material would exceed, in the long run, the following values only in 1 case in 20:

$$\text{Reproducibility} = 0.455 \sqrt{x}^*$$

*where x is the average value of two results in ug/g.

9.2 Bias. The bias of this test method varies with concentration, as shown in Table 2:

Bias = Amount found - Amount expected

TABLE 1

REPEATABILITY AND REPRODUCIBILITY FOR CHLORINE IN USED OILS BY MICROCOULOMETRIC TITRATION

Average value, ug/g	Repeatability, ug/g	Reproducibility, ug/g
500	69	228
1,000	137	455
1,500	206	683
2,000	274	910
2,500	343	1,138
3,000	411	1,365

TABLE 2

RECOVERY AND BIAS DATA FOR CHLORINE IN USED OILS BY MICROCOULOMETRIC TITRATION

Amount expected, ug/g	Amount found ug/g	Bias, ug/g	Percent bias
320	312	-8	-3
480	443	-37	-8
920	841	-79	-9
1,498	1,483	-15	-1
1,527	1,446	-81	-5
3,029	3,016	-13	0
3,045	2,916	-129	-4

1. Gaskill, A.; Estes, E.D.; Hardison, D.L.; and Myers, L.E. Validation of Methods for Determining Chlorine in Used Oils and Oil Fuels. Prepared for U.S. Environmental Protection Agency, Office of Solid Waste. EPA Contract No. 68-01-7075, WA80. July 1988.

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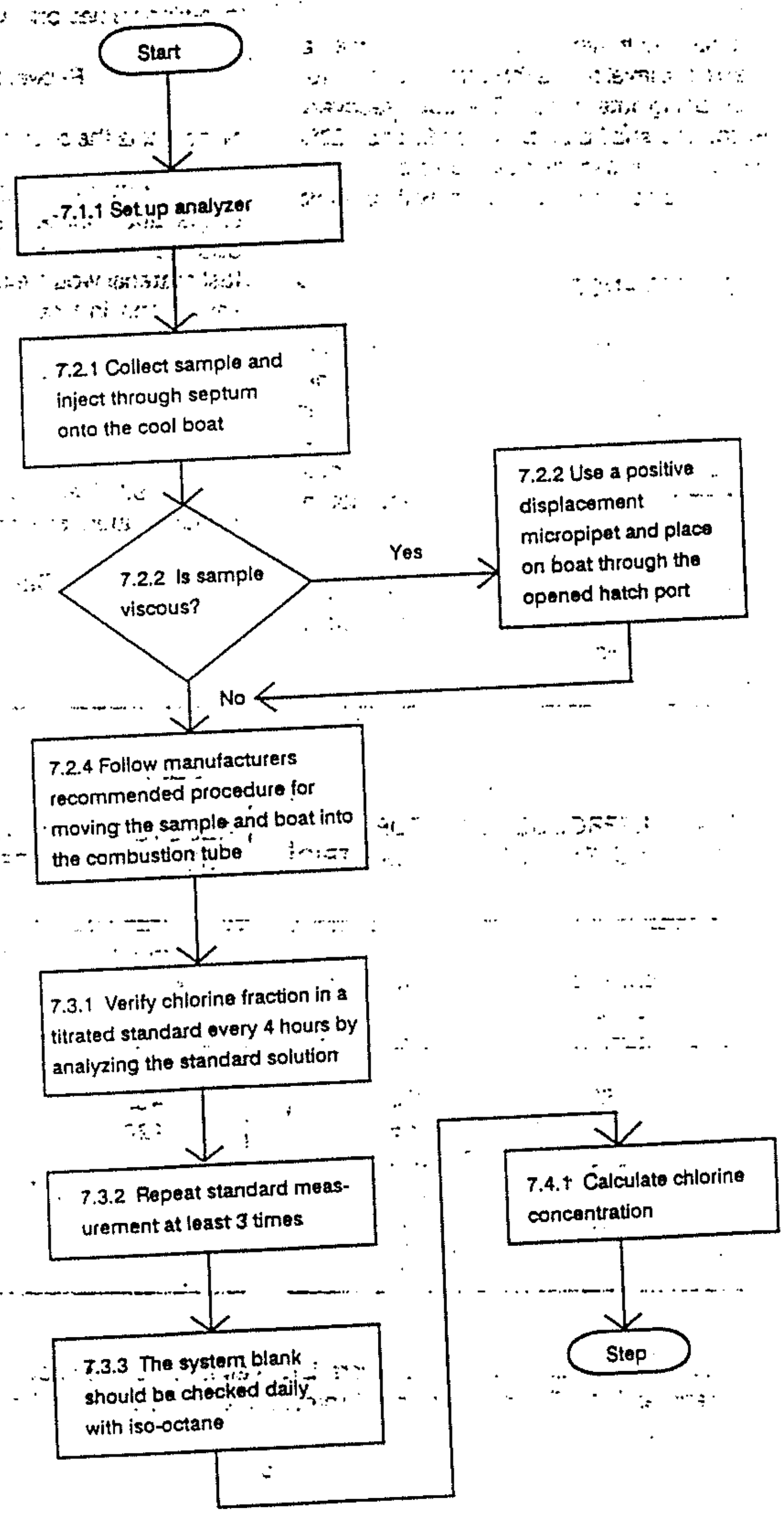
OXIDATIVE COMBUSTION AND MICROCOULOMETRY



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SECTION 6.0

COMPLIANCE WITH SPECIFIC REGULATIONS

6.0 COMPLIANCE WITH SPECIFIC REGULATIONS

6.1 LAND DISPOSAL RESTRICTIONS

Federal land disposal restrictions (LDR) are designed to minimize the volume of hazardous waste disposed to land and to foster additional treatment or recycling prior to disposal. Federal hazardous wastes that are destined for land disposal must meet the applicable treatment standard before the material can be disposed of to land. Treatment standards may be expressed as a specific treatment technology, a constituent concentration in a waste or waste extract, or both.

6.1.1 Notifications/Certifications Received from Generators

Most federal hazardous wastes received at PRC could be subject to LDR. Each shipment of a restricted waste transported to PRC should be accompanied by a notification of whether the material meets the applicable treatment standards. This notification should also include the following information:

- EPA hazardous waste codes.
- Corresponding treatment standards and all applicable prohibitions.
- Manifest number associated with the shipment.
- Waste analysis data, where available.

If the waste meets the applicable treatment standard, a certification statement should be included.

Notifications and certifications must be retained for at least 5 years.

6.1.2 Wastes Generated by PRC

All outgoing shipments of material classified as federal hazardous waste should be accompanied by an LDR notification as described in Section 6.1.1. This applies to wastes destined for further treatment as well as those destined for disposal. The exception is waste which is hazardous based solely on TCLP constituent criteria; treatment standards have not yet been promulgated for those wastes.

Each shipment of waste leaving the facility should be tested using the appropriate procedures. Analytical tests will be performed by a state-certified laboratory. For wastes with treatment standards expressed as concentrations in the waste extract, analysis should be performed on the extract obtained using the TCLP outlined in 40 CFR, Part 268, Appendix I.

6.2 BENZENE NESHAPS

The NESHAPS regulations will apply to the streams that PRC receives from a facility subject to the NESHAPS regulation (chemical manufacturing plants, coke by-product recovery plants, and petroleum refineries). A compliance schedule for the plant is as follows:

1. Streams which PRC receives from facilities subject to NESHAPS will be tested for benzene concentrations following the requirements in the NESHAPS rule. Estimated completion date - 7/26/91.
2. The volume of material received from NESHAP sources will be estimated. Estimated completion date - 7/26/91.
3. Annual benzene quantity through facility from NESHAPS sources will be calculated. A draft report that summarizes the regulatory status of each stream will be prepared. Estimated completion date - 9/1/91.
4. Determination of status under this regulation. Estimated completion date - 10/1/91.
5. Final report on facility status. Estimated completion date - 12/21/91.